



May 29, 1986

Mr. R.L. Boiselle, P. Eng.
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Dear Mr. Boiselle:

Enclosed are the final reports in ^{duplicate}~~triplicate~~ and payment claim in fulfillment of DSS Contract Serial No. OSQ85-00154.

The total package includes:

1. Report by M.E. Wiseman on Laurentian University work "A Comparative Study of Three Methods of Reducing Acid Generation and Metals Release from Waste Rock and Tailings".
2. Executive summary by R.E. Michelutti and report by Boojum on "Developing a Technique for Reclamation of High Iron Sulphur Tailings by Establishing a Marshland".
3. Falconbridge final claim report.

I trust that all is in order. Please call me if there are any questions.

Yours truly

R.E. Michelutti
Supervisor Environmental Control

REM:wb
Encl.

bcc: A.G. Cecutti
M.E. Wiseman



**A COMPARATIVE STUDY OF THREE METHODS
OF REDUCING ACID GENERATION AND METALS
RELEASE FROM WASTE ROCK AND TAILINGS**

FALCONBRIDGE

**A COMPARATIVE STUDY OF THREE METHODS
OF REDUCING ACID GENERATION AND METALS
RELEASE FROM WASTE ROCK AND TAILINGS**

Work on this project was conducted under the auspices of the Canada
Centre for Mineral and Energy Technology, Energy, Mines and
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MICROMEDIA

EXECUTIVE SUMMARY:

This research was initiated to evaluate three methods of economically controlling acid generation and metals release from waste rock and tailings. The first method utilized inexpensive chemicals to control bacterial and/or chemical oxidation. Chemicals evaluated include the bactericides sodium lauryl sulphate and Promac, as well as chemical control with hydrated lime, calcium phosphate monobasic, and apatite.

The second method involves covering the tailings with a four foot cover of waste rock and digested sewage sludge. The combination of these two waste products should decrease water and air penetration to the tailings, improve pH, decrease Thiobacillus ferrooxidans (bacteria) activity and provide a substrate for revegetation. These factors should decrease chemical and bacterial oxidation of both the tailings and waste rock, decreasing metal and acid loads to the environment.

The third method involves covering the tailings with a four foot cover of municipal garbage, sewage and their cover (in this case waste rock) to prevent the garbage from blowing and provide a substrate for revegetation. Rotting garbage and sewage will generate an anoxic, reducing environment which can prevent oxidation of tailings and reduce metal and acid loads downstream.

During 1985, the local strain of T. ferrooxidans bacterium was isolated and cultured. Calcium phosphate monobasic, hydrated lime and sodium lauryl sulphate were evaluated in 9K growth media and shake flasks to

determine their potential to decrease bacterial and/or chemical oxidation of sulphides, and to determine required concentrations for effective treatment. Results from these experiments, in conjunction with literature data, were utilized to establish leach columns and further evaluate their effects under more natural and realistic conditions. The leach column experiments are ongoing. It is proposed to field test the best treatment chemical in the Fraser Mine area in 1986, assuming an effective control is found.

Revegetation experiments were initiated in 1985 utilizing waste rock and sewage on the Fecunis Tailings Pond. Twenty-four plots with variable sewage, limestone, fertilizer and seed mixtures were established and will be evaluated in 1986.

Experiments conducted to improve the persistence of sodium lauryl sulphate (bactericide), were unsuccessful.

Test plots were constructed on the Fecunis Tailings Pond to evaluate the effectiveness of garbage, waste rock and sewage covers to reduce acid and metals release from the tailings. Due to the late start, data collection will begin in 1986.

ACKNOWLEDGEMENTS:

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A COMPARATIVE STUDY OF THREE METHODS
OF REDUCING ACID GENERATION AND METALS
RELEASE FROM WASTE ROCK AND TAILINGS

INTRODUCTION:

Historically, much of the research for reclamation of mine-mill sites has centered on mill tailings reclamation. It was generally thought that the coarse waste rock was relatively innocuous. Tailings, in addition to being aesthetically worse than waste, was assumed to leach faster due to the smaller particle size and higher sulphur and metal concentrations. As a result, tailings is impounded by dams, while waste rock, until recently, has been widely used in roads, railroad beds, yard and dams.

Recent preliminary data from the Strathcona Creek-Moose Lake water treatment system indicates that the largest percentage (50-80%) of the metal load to the environment is leached from mine-mill yards, roads and railroad beds, with less than 50% from the active and abandoned tailings pond. As a result there are major capital and operating expenses associated with the control and treatment of the natural drainage from waste rock disposal areas.

The reasons for the high contaminant contribution from waste rock have recently been realized. Coarse waste rock has large pore spaces to permit relatively high oxygen and water permeability through its entire depth. Oxidation is not limited to rock surfaces. Bacteria, which have been proven to accelerate acid generation and metals dissolution, can colonize throughout the waste rock. Bacterial activity is often

accelerated by the presence of moisture, nutrients, fluctuating water tables, and heat generated by oxidation. In contrast, tailings oxidation is primarily a surface phenomenon (several centimeters), especially in active tailings which remains wet. Oxygen and water permeability rates are much lower than in the waste rock, and bacteria have less opportunity to flourish. The impoundment of tailings limits exposure to water table fluctuations, organics, nutrients, and heat generation. Finally, with today's improved metals recoveries, the metal content of tailings can be surprisingly close to that of waste rock. Therefore, when evaluating methods to minimize mine-mill leachate impact on the environment, it is essential to control acid generation and metals dissolution from waste rock as well as tailings.

Tailings treatment systems have traditionally been designed to treat only tailings effluents, with diversion of as much natural drainage water as possible away from the system. This is done to minimize the volume of water contaminated and to minimize treatment costs.

Unfortunately, large volumes of waste rock have historically and are still being utilized for road, yard and railroad bed construction. This has resulted or will result in significantly larger drainage areas, water volumes and contaminant loads that require treatment. This problem can create the need for new treatment systems or the expansion of existing systems during the life of the operation. Assuming long term abandonment strategies are developed for the waste rock, the large capital and operating costs of these systems or system modifications are therefore of short term benefit only. It would be preferable to develop cost effective technology to inhibit or stop the waste rock from leaching as the waste piles develop or are in use. In light of the cost

of new or expanded treatment facilities, short term waste rock treatment strategies may be more economical while the mine is in operation, or on a long term basis. Alternately, such techniques would be utilized until a permanent abandonment strategy for the piles has been determined.

While a considerable quantity of research is available on methods of accelerating leaching processes, research into reversing these processes is less developed. Some significant short term reductions in metal and acid generation have been realized in Southern coal spoils, and research now is attempting to extend the effectiveness of these treatments.

Falconbridge Limited in conjunction with Canmet and Laurentian University, is presently evaluating seven chemical applications in the laboratory. Each of these chemicals, if effective, is inexpensive enough that short term benefits which would necessitate annual chemical application, are significantly more economical than new or expanded treatment facilities.

The reclamation of waste rock and tailings with thin vegetative covers has not been proven to significantly reduce acid or metal generation in the short term. Research is required to economically reduce metal and acid generation at the source under the extremes of the Northern Ontario climate. Both short term and long term strategies are required if waste rock is to be utilized for construction purposes. Ideally, elimination of these leachates at the source could present a more cost effective alternative to the costly water collection and treatment systems now in use.

CONCEPTS:

The most economical way to reclaim tailings and/or waste rock is to utilize available waste products. The use of municipal garbage, sewage, and waste rock in conjunction with chemical treatment and sound management practices is proposed in this multi-faceted approach to waste rock and tailings treatment. Research into the effectiveness of the three techniques proposed below is required.

1) Tailings, Waste Rock and Sewage

Vertical migration of acidity and metals in tailings to the surface during dry periods makes direct seeding difficult and normally requires extended maintenance. Also, in most situations a thin gravel or topsoil overburden placement is very costly, and therefore prohibitive as well as ineffective in controlling leachates in the short term. The most cost effective way to cover abandoned tailings is to utilize available waste materials. As it turns out, the use of a broad surface area such as tailings is a cost effective way to dispose of waste rock, by limiting bulldozer time. Waste rock on tailings has several advantages. A thick three to four foot cover stabilizes the tailings with a safe covering which prevents dusting and erosion. The waste rock will reduce tailings capillary action by preventing the surface from drying out. Dry abandoned tailings leach faster than wet active tailings. The large pore spaces in waste rock will prevent leachate from waste rock or tailings from migrating to the surface via capillary action, and in fact should drain any leachates away from the root zone. Vertical migration of

leachates in thin organic covers such as topsoil, can kill plant life. To further reduce oxidation in the waste, municipal sewage can be spread on the waste to fill surface pore space, reducing oxygen and water permeability to the waste and tailings. The sewage provides a substrate for reclamation with plants. The sludge and plants should also moderate soil and water pH, reducing soluble ferric (Fe^{3+}) ion concentrations and decreasing Thiobacillus ferrooxidans (bacteria) activity, which also will reduce leaching. Finally the combining of both acid generating materials, tailings and waste rock, into one location is a sound management practice.

2) Municipal Garbage

The use of municipal garbage and/or mine-mill domestic garbage in a similar manner to the waste rock in the above concept is proposed. This is advantageous where waste rock is in limited supply. Municipalities can reduce garbage disposal costs, especially with respect to selection and development of new garbage disposal sites. Mining companies can benefit by municipal reclamation of their tailings in a cooperative and mutually beneficial arrangement.

While utilization of garbage covers have been evaluated in the laboratory, they generally have evaluated relatively thin covers with direct seeding. Composted garbage has been shown to be an effective reclamation cover. However, to compost garbage requires sorting, removal and disposal of metal and non compostables, shredders, grinders, crushers, mixers and the blending of this material with wood chips and/or organics such as sewage sludge. The

sludge generally requires dewatering and "maturing". The cost of this is prohibitive (A. Jongejan, 1981).

Nevertheless, raw domestic municipal garbage does provide a cheap available overburden material which, with a thick enough layer, can limit oxygen and water penetration to the tailings. Garbage particle size is such that it will prevent vertical migration of contaminants to the surface. As the garbage decays, as with wetland technology, an anoxic, reducing environment should be created at the tailings surface, effectively stopping acid and metal generation. In fact, co-disposal of domestic garbage and heavy metal bearing acids is a common and acceptable disposal practice in the United Kingdom (Finch, 1986). Since the raw garbage does not provide an adequate substrate for vegetation, overburden is required for planting. This could be gravel, topsoil or mine waste rock, and since the garbage must be landfilled this does not result in additional costs for reclamation.

3) Chemical Treatment

Sewage and/or garbage cannot be utilized in all areas where waste rock is found, such as mine yards or roads. Therefore, a practical technique such as a chemical treatment is required to prevent leaching until permanent abandonment techniques can be determined and applied.

PHASE 1 - FIELD WORK

PURPOSE: To verify the presence of Thiobacillus ferrooxidans in the Onaping Area waste rock and to isolate the indigenous strain. Since individual strains of bacteria will adapt to conditions at any particular site, it was important to isolate the local strain, one which was tolerant and/or adapted to the existing pH and heavy metal concentrations.

METHOD: In July, 1986, water samples from five locations in the Onaping Area were sampled. Utilizing standard membrane filtration techniques and 9k media, the strain of Thiobacillus ferrooxidans found in the Onaping Area was isolated. All sites sampled had the bacterium present.

PHASE 2 - LABORATORY STUDIES

The second phase of this research involved laboratory experimentation. The purpose of the laboratory work is twofold. While several researchers have evaluated various methods of decreasing oxidation in waste rock, (Kleinmann, 1981; Watkin, 1983; Stiller, 1983; Fox, 1983) different methodologies, wastes and environmental conditions existed at Falconbridge. The following experiments were performed to directly compare the different treatments as well as to estimate the amount of a particular treatment required to control Ni-Cu-Fe and acid generation from waste rock found in the Sudbury Area.

EXPERIMENT #1 - Impact of Thiobacillus ferrooxidans on the
Leaching Rate of Ni-Cu Sulphide Mill Feed
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PURPOSE: To evaluate the effect of an inoculum of Thiobacillus ferrooxidans on the leaching rate of a Cu-Ni-Fe sulphide mill feed in shake flasks. This experiment will evaluate whether or not the local bacterial strain can significantly accelerate oxidation rates and increase Ni, Cu, Fe and acid release to the environment. It will show whether or not control of local bacterial oxidation can be useful in pollution control.

METHOD: Fifty grams of Strathcona Mill feed were added to two 250 ml Erlenmeyer flasks containing 100 mls of distilled water. Details of the mill feed are itemized in Table 1.1. The flask contents were adjusted to pH 2.5 with sulphuric acid and autoclaved.

One flask was then inoculated with one ml of a Thiobacillus ferrooxidans suspension (Optical density = 0.843). The other flask remained as a sterile chemical leach control. Nickel, copper, ferric iron, and pH were monitored periodically for 120 days. Periodic acid additions were required in the sterile control to maintain the same 2.5 pH as the inoculated flask.

Since only a general trend was looked for, and with existing cost restraints, no replicates were performed.

TABLE 1.1 - Strathcona Mill Feed

Nickel	1.10%
Copper	0.09%
Sulphur	9.7%
Iron	21.0%
Magnesium	0.037%
Nitrogen (as N)	30 mg/kg
Phosphate	0.25 mg/kg

TABLE 1.2 - Impact Of T. ferrooxidans On The Leaching Rate Of Ni-Cu-Fe Sulphide Mill Feed - Data

Time (days)	pH		Fe ³⁺ (mg/L)		Ni (mg/L)		Cu (mg/L)	
	Control	Inoculated	Control	Inoculated	Control	Inoculated	Control	Inoculated
0	8.3	8.12	4	2	17.0	21.5	.16	.15
1	4	3.30						
2	3.89	3.34	8	12	30.0	33.8	1.32	1.99
7	2.96	2.85						
15	3.19	3.12	29	28	58.0	44	1.85	2.41
21	4.15	2.89						
28	4.11	3.57						
35	3.79	2.46	4	120	61.5	95	0.09	1.96
55	4.40	2.94						
84	4.37	2.69						
85	2.91	2.56						
86	2.77	2.52	62	400	69.0	135	0.48	6.2
93	3.55	2.77	67	196	74.0	140	0.26	6.8
108	3.47	2.63	81	231	78.0	174	0.06	
120	3.13	2.26	35	515	81.0	196	0.20	13.7
153					45.0	106	0.03	7.0

RESULTS: As expected, concentrations of acid, copper and iron were considerably greater in the inoculated flask than in the control flask (Table 1.2).

CONCLUSIONS: Elimination or reduction of local populations of Thiobacillus ferrooxidans can significantly reduce release

of nickel, copper, iron and acid generation in Ni-Cu-Fe sulphides.

EXPERIMENT #2 - Evaluation of Bactericidal Effect of Sodium Lauryl Sulphate on Mine - Environment Isolates of Thiobacillus ferrooxidans at 20°C.

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PURPOSE: To evaluate bactericidal effect of sodium lauryl sulphate (S.L.S.) on mine-environment isolates of Thiobacillus ferrooxidans at an incubation temperature of 20°C in shake flasks.

METHOD: A 500 mg/L stock solution of sodium lauryl sulphate $(CH_3)(CH_2)_{10}CH_2OSO_3Na$, (sodium dodecyl sulphate) was prepared with reagent grade BDH chemical powder.

After carefully mixing, so as to prevent foaming, a portion of this stock solution was poured into a 260 ml Erlenmeyer flask and autoclaved at 121°C for 25 minutes.

The following concentrations of S.L.S. were evaluated: 5, 10, 25 and 50 mg/L. Both inoculated and uninoculated controls were also run. Appropriate volumes of the stock S.L.S. solution were aseptically delivered to duplicate 250-ml sample flasks containing 42 ml of sterile 9k medium. Three ml of a logarithmic phase 9k culture ($Fe^{+++} = 6322$ ppm) were added to the sample flasks and an appropriate volume of sterile distilled water was added to

provide a final sample volume of 50 ml. The 50 ml 9k chemical control was not inoculated and was included only to determine the rate of chemical oxidation of Fe^{++} to Fe^{+++} . The amount of Fe^{+++} produced through chemical oxidation of the substrate was subtracted from the total reading at each sampling period to determine bacterial oxidation rates. All samples were incubated at 20°C in an Eberbach reciprocating shaker at medium speed.

RESULTS:

No growth was detected in the S.L.S. treated samples after 200 hours. Normal growth occurred in the control sample replicates with an average generation time of 19.7 hours. One replicate from each of the S.L.S. treated samples was centrifuged and the cells washed and collected. These cells, which were visible under microscopic examination, were then inoculated into fresh 9k medium to check for viability. No growth occurred within eight days indicating that the range of S.L.S. used was bactericidal.

After 120 hours of growth, the chemical oxidation rate was determined. Chemical oxidation occurred at the following rate:

$$y = 0.0047 x + 4.922$$

where $y = \ln \text{Fe}^{3+}$ concentration in mg/l

$x =$ time in hours

$r = 0.964$ at the 95% confidence limit

This is roughly equal to 154 mg/L Fe^{3+} per day.

CONCLUSIONS: A S.L.S. concentration of 5-50 mg/L is bactericidal for the mine isolates of Thiobacillus ferrooxidans tested.

Chemical oxidation rates represented less than 4% of the total oxidation of Fe^{3+} at peak biological oxidation rates.

Further research is required to determine the minimum concentration of S.L.S. bactericidal for Thiobacillus ferrooxidans.

EXPERIMENT #3 - Determination of the Minimum Bactericidal Concentration of Sodium Lauryl Sulphate at 20°C.

PURPOSE: To determine the minimum bactericidal concentration of sodium lauryl sulphate on mine-environment isolates of Thiobacillus ferrooxidans at 20°C.

METHOD: The same S.L.S. stock solution from Experiment #2 was utilized. The following S.L.S. concentrations were evaluated: 0, 1, 2, 3 and 4 mg/L. The chemical oxidation rate from Experiment #2 was assumed constant and was not re-evaluated.

Appropriate volumes of the stock S.L.S. solution were aseptically delivered to the 250 ml duplicate sample flasks containing 43 ml of sterile 9k medium. Three ml aliquots of a logarithmic phase 9k culture ($\text{Fe}^{+++} = 6149$

ppm) were added to the sample flasks and an appropriate volume of sterile distilled water was added to provide a final sample volume of 50 ml. All samples were incubated at 20°C in an Eberbach reciprocating shaker at medium speed.

RESULTS:

Results of this experiment are summarized in Table 3.1. These results suggested that S.L.S. concentrations greater than 3 mg/L were bactericidal. In order to check this, the contents of one replicate (50 ml 9k) were centrifuged and the cells washed and collected. These cells were then inoculated into fresh 9k medium to check for viability. Cells from the 3 mg/L sample grew within three days, while the cells from the 4 mg/L sample showed no signs of growth after seven days.

TABLE 3.1 - Growth of Thiobacillus ferrooxidans in 0 to 4 mg/L S.L.S. at 20°C.

<u>S.L.S.</u> <u>(mg/L)</u>	<u>Lag (hrs)</u>	<u>Average</u> <u>Generation</u> <u>Time (hrs)</u>	<u>Maximum</u> <u>Fe⁺⁺⁺</u> <u>(mg/L)</u>
1	50	22.8	5580
1	50		5580
2	150	23.4	4426
2	150		4478
3	N.G.*		377
3	N.G.		345
4	N.G.		345
4	N.G.		362
0	0	20.7	5507
0	0		5272

* N.G. = no growth

CONCLUSIONS: Sodium lauryl sulphate was bactericidal at concentrations at or above 4 mg/L. A concentration of 3 mg/L is either bacteriostatic for the isolate tested or the lag is greater than 300 hours. At 1 mg/L S.L.S., the initial lag was found to be 50 hours with an average generation time of 22.8 hours. At 2 mg/L S.L.S. the lag was 150 hours with an average generation time of 23.4 hours. The control sample, which had no S.L.S. added, did not have a lag time and had an average generation time of 20.7 hours. It is further recommended that S.L.S. be evaluated under conditions similar to the field such as in column leach experiments.

EXPERIMENT #4 - Determination of the Temperature Effect on the Minimum Bactericidal Concentration of Sodium Lauryl Sulphate on Thiobacillus ferrooxidans.
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PURPOSE: To examine the minimum bactericidal concentration of sodium lauryl sulphate (S.L.S.) on mine environment isolates of Thiobacillus ferrooxidans at 12°C.

INTRODUCTION: Bacteria can have a wide range of responses to temperature. For example, lower temperatures can slow metabolic rates which means that the bactericidal concentration of an individual chemical may be much higher at lower temperatures. Therefore, an evaluation of the effect of temperature on the bactericidal concentration of

S.L.S. was required before its potential use in northern climates.

METHOD:

The same stock solution from Experiment #2 was utilized. The following S.L.S. concentrations were evaluated: 0, 1, 2, 3 and 4 mg/L. The same chemical oxidation rate from Experiment #2 was assumed and was not re-evaluated.

Appropriate volumes of the stock S.L.S. solution were aseptically delivered to the 250 ml duplicate sample flasks containing 43 ml of sterile 9k medium. The control sample (0 mg/L S.L.S.) was not replicated. Three ml aliquots of a logarithmic phase 9k culture were delivered to the sample flasks and an appropriate volume of sterile distilled water was added to provide a final sample volume of 50 ml. All samples were incubated at 12°C in an Eberbach reciprocating shaker at medium speed.

RESULTS:

Results from this experiment are summarized in Table 4.1.

The results again suggest that S.L.S. concentrations greater than 3 mg/L were bactericidal, as in the 20°C experiment. The cells from a 3 and 4 mg/L replicate were collected by centrifugation and inoculated into fresh 9k medium to check for viability. Growth was detected in the sample collected from the 3 mg/L S.L.S. flask within 3 days. No growth was detected after seven days in the sample prepared from the 4 mg/L S.L.S. flask.

TABLE 4.1 - Effects of 0-4 mg/L S.L.S. on Generation Time and Fe^{3+} Release by *T. ferrooxidans* at 12°C .

S.L.S. (mg/L)	Lag (hrs)	Average Generation Time (hrs)	Maximum Fe^{+++} (mg/L)
1	20	35.2	6063
1	20		6219
2	70	43.8	6188
2	70		5927
3	N.G.*		337
3	N.G.		369
4	N.G.		306
4	N.G.		380
0	0	31.6	6272
0	0		5272

* N.G. = no growth

CONCLUSIONS: The results are similar to the S.L.S. experiment conducted at 20°C . The bactericidal concentration was determined to be 4 mg/L for 9k cultures of a mine-environment isolate of *Thiobacillus ferrooxidans* at an incubation temperature of 12°C . A concentration of 3 mg/L is either bacteriostatic for the isolate tested or the lag is greater than 300 hours. At 1 mg/L S.L.S. the lag appeared to be 20 hours and the 2 mg/L sample had a 70 hour lag. This was approximately half the lag seen at similar concentrations of S.L.S. for the 20°C experiment. However, as in the 20°C experiment, no growth occurred at 4 mg/L S.L.S., and therefore the difference in lag periods is of academic

interest only.

While a temperature effect was observed, it was not a significant difference, and as such will not be a major concern in the field.

EXPERIMENT #5 - Evaluation of the Impact of Calcium Hydroxide and Calcium Hydroxide Precipitates on Chemical and Bacterial Oxidation of Copper-Nickel-Iron Sulphides.
.....

PURPOSE: To evaluate the impact of calcium hydroxide and calcium hydroxide precipitates on chemical and bacterial oxidation of copper-nickel-iron sulphide under neutral and acidic conditions.

INTRODUCTION: Lime has historically been applied to waste rock by Falconbridge Limited to reduce metal and acid generation. This is a simple and relatively inexpensive method to reduce acid and metal generation, but has a short term effect. An application rate of 5 tons/acre on a 25 ft. column of waste rock is roughly equivalent to 88. mg/kg. However, since we were starting with fresh alkaline ore, and in an effort to accelerate the experiment, lower lime additive rates were evaluated. This experiment will be utilized for economic and effectiveness comparison to other treatments and will determine the stability and impact of lime precipitates.

PROCEDURE: Fifty grams of mill feed were added to duplicate 250 ml

Erlenmeyer flasks. Appropriate amounts of a 1000 mg/L $\text{Ca}(\text{OH})_2$ solution were added to the flasks to give the following concentrations: 0, 4.4, 8.8, 16 and 35 mg/kg. Solutions were made up to 100 mls and autoclaved. The flasks were placed on a reciprocating shaker at medium speed and room temperature.

After 71 days, it was decided to accelerate the experiment by the addition of concentrated sulphuric acid. Flask pH's were gradually lowered to 3.0 and inoculated with Thiobacillus ferrooxidans. This was done in an attempt to determine if lime precipitates or complexes had any inhibitory effect on bacterial oxidation.

Only pH was monitored until day 90. Nickel, copper, iron and pH concentrations were monitored on day 90 and 112.

RESULTS: Initial pH's were 8.5 to 9. All samples, with the exception of the 35 mg/kg sample had stabilized near pH7 within one week. The 35 mg/kg sample took approximately three weeks to stabilize. After 10 weeks, there was no significant change in solution pH, and accelerated acidification was begun. (Figure 5.1)

Table 5.1 summarizes the acid additions required to bring the flask pH's below 3.0.

TABLE 5.1 - Acid Required To Obtain pH 3.0

mg/kg Ore	Lime Solution Concentration (mg/L)	Equivalent Addition Rate on 25 ft. Waste Rock Column lb/acre	Milliequiv. of H_2SO_4
0	0	0	37.2
4.4	2.2	250.	37.7
8.8	4.4	500.	39.2
16	8.8	1000.	38.5
35	17.6	2000.	38.6

It was interesting to note that the bulk of the alkalinity was natural, rather than from the added lime. Further analysis of this phenomenon are presented later.

Fe^{3+} concentrations increased to an average 260 mg/L during the first 90 days, of which 20 days were under acidic conditions. Between day 90 and 111, Fe^{3+} concentrations increased to an average 1532 mg/L, indicating bacterial oxidation. Bacterial oxidation occurred in seven of the ten replicates (Table 5.2). This suggests that lime or lime complexes or precipitates has no effect on biological oxidation. The reason for low biological oxidation in three of the ten replicates is unknown and is not necessarily related to lime addition. Leaching of nickel and copper (Tables 5.3, 5.4) did not correlate with iron oxidation rates and bacterial activity. Copper concentrations decreased with increasing lime concentrations, possibly due to adsorption of copper by the bacteria. Nickel concentrations increased in all

flasks, including those with low iron concentrations and bacterial activity, suggesting that the low pH was responsible for chemical oxidation of nickel.

It was unfortunate that these experiments could not have been continued to evaluate long term impact on nickel and copper leachates due to bacterial oxidation and acid and Fe^{3+} generation. A long term evaluation of the effects of lime in column leach experiments which more closely resemble field situations is recommended.

TABLE 5.2 - Iron (Fe^{3+}) Concentrations vs Time at 0 to 35 mg/kg Lime Application Rates

Time (Days)	Control 0 mg/kg		4.4		8.8		16		35	
	A	B	A	B*	A*	B	A	B*	A	B
90	132	166	141	157	494	278	258	339	501	137
103	527	489	429	182	209	711	1766	238	452	1479
112	1377	1328	1286	28	374	1913	1557	447	1113	2153

*No bacterial oxidation

TABLE 5.3 - Copper Concentrations vs Time at 0 to 35 mg/kg Lime Application Rates

Time (Days)	Control 0 mg/kg		4.4		8.8		16		35	
	A	B	A	B*	A*	B	A	B*	A	B
90	69	66	88	69	86	75	75	220	210	87
112	22	22	18	40	30	3	14	104	139	12

**TABLE 5.4 - Nickel Concentrations vs Time at 0 to 35 mg/kg
Lime Application Rates**

Time (Days)	Control 0 mg/kg		4.4		8.8		16		35	
	A	B	A	B*	A*	B	A	B*	A	B
90	588	613	610	540	725	625	660	575	570	545
112	740	790	790	940	940	750	1020	800	840	680

*No bacterial oxidation based on Fe^{3+} concentrations.

**EXPERIMENT #6 - Evaluation of the Effects of Calcium
Phosphate Monobasic and its Precipitates
on the Oxidation of Ni-Cu-Fe Sulphides
Under Neutral and Acidic Conditions**

PURPOSE: To evaluate the ability of calcium phosphate monobasic to inhibit the oxidation of Ni-Cu-Fe sulphides under neutral and acidic conditions.

METHOD: One liter of a 10,000 ppm stock solution of $Ca(H_2PO_4)_2 \cdot H_2O$ was made up. Fifty grams of Strathcona Mill feed (Table 1, Experiment 1) were added to each of nine, 250 ml culture flasks and autoclaved. The following wide range of $Ca(H_2PO_4)_2 \cdot H_2O$ concentrations were evaluated: 200, 600, 1000, and 2000 mg/L. An appropriate amount of sterile distilled water was then added to the flasks so that the final liquid volume was 100 ml. A sterile control with no calcium phosphate monobasic was also run.

The control sample was found to have a pH of 8.6. The addition of the $Ca(H_2PO_4)_2 \cdot H_2O$ tended to decrease the sample pH. Murakami (1963) reported that in order for the

phosphate precipitates to coat the sulphide particles, the pH should be neutral. Accordingly, the pH of all the samples was adjusted to pH 7.0 using either 1N H_2SO_4 or 30% KOH. Samples were taken for Fe^{3+} , Ni, Cu and pH. Approximately 1.5 ml of sample was withdrawn from each replicate. This supernatant was filtered through a Millipore (HAWP #01300) 0.45 μm filter to remove particulate matter. After filtering, 1 ml of the sample was delivered to a plastic sample vessel to which 9 ml of distilled water had been added to give an initial sample dilution of 1/10. This sample was later analyzed for Cu and Ni by atomic adsorption. A portion of the remainder of the sample was then tested for Fe^{+++} using the thiocyanate method of Kolthoff et al, 1969. Adsorbance was measured at 480 μm with a Novaspectrophotometer.

After 100 days of inoculation, there was little change in pH in the samples. As in Experiment #5, it was decided to accelerate the experiment by gradually lowering and stabilizing the pH with concentrated sulphuric acid to a pH of approximately 3.5.

On day 121 the samples were inoculated with 1 ml of a stock (O.D. = 14.76) suspension of a mine environment isolate of Thiobacillus ferrooxidans. If the phosphate had been effective in forming a protective coating (precipitate) around the sulphide particles, then presumably only the control (0 g phosphate added) would

show signs of biological oxidation after the inoculation. Samples continued to be taken for pH, Ni, Cu and Fe^{3+} .

RESULTS:

The pH results are summarized in Table 6.1 and Figure 6.1.

TABLE 6.1 - Initial pH After $\text{CaHPO}_4 \cdot \text{H}_2\text{O}$ Additions and Acid Required to Reach pH 3.

Phosphate Concentration (mg/L)	Initial pH	Acid Required To Reach pH 3 After Day 100 (meq)
0 (control)	8.6	37.0
200 (200 mg/kg)	7.09	38.7
600 (1200 mg/kg)	5.59	40.1
1000 (2000 mg/kg)	5.17	42.7
2000 (4000 mg/kg)	4.60	56.3

There was no measurable Fe^{3+} produced prior to the acidification. Nickel and copper concentrations were also low. Subsequent to the addition of the acid, dramatic increases in dissolved nickel, copper and Fe^{3+} were observed. The nickel, copper and iron data are summarized in Tables 6.2 to 6.4 and Figures 6.2 to 6.4.

TABLE 6.2 - Fe³⁺ Concentrations vs Time and Calcium Phosphate Concentration

Concentration of Ca(HPO ₄) ₂ ·H ₂ O									
Time (Days)	0 mg (control)	200 mg		600 mg		1000 mg		2000 mg	
		A	B	A	B	A	B	A	B
0	0	0	0	0	0	0	0	0	0
7	0	0	0	0	0	0	0	0	0
16	0	0	0	0	0	0	0	0	0
30	0	0	0	0	0	0	0	0	0
Samples Acidified									
121	575	638	588	465	469	383	606	195	111
Bacteria Added									
123	339	327	303	325	229	140	139	50	79
134	438	187	613	199	230	114	149	353	165
142	490	440	819	288	435	395	143	195	205
151	335	481	340	202	387	272	246	411	280
175	100	233	76	149	278	128	49	53	34

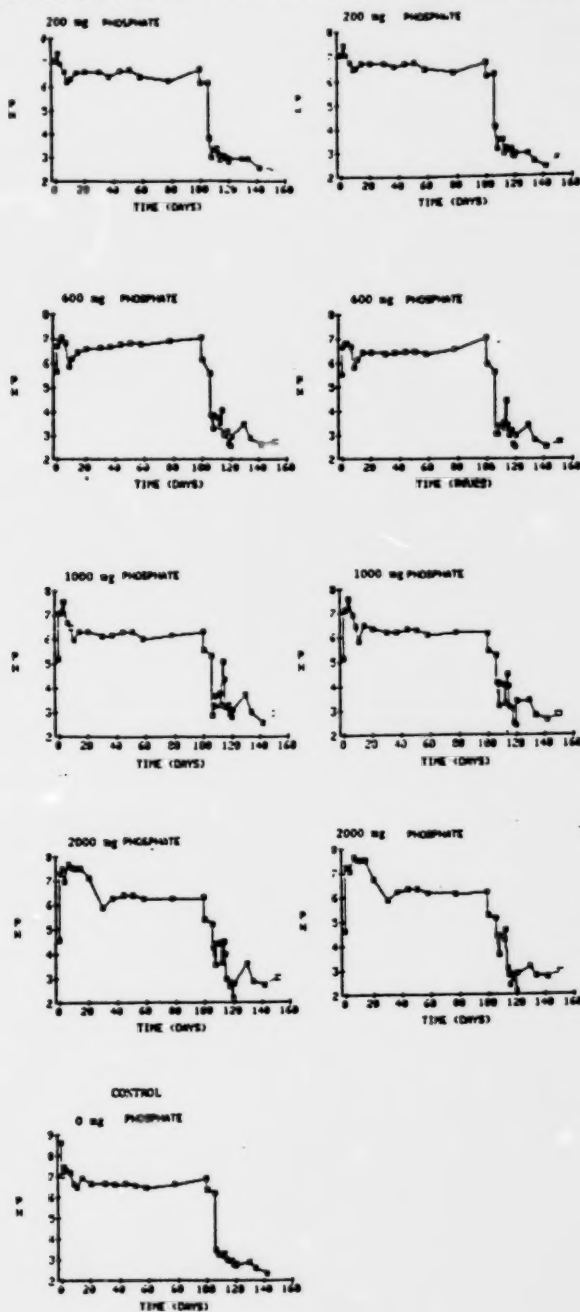
TABLE 6.3 - Copper Concentration (ppm) vs Time and Calcium Phosphate Concentration

Calcium Phosphate Concentration									
Time (Days)	0 mg (control)	200 mg		600 mg		1000 mg		2000 mg	
		A	B	A	B	A	B	A	B
0	1.4	0.5	0.8	1.7	1.7	1.9	1.9	3.6	4.0
30	0.9	0.3	0.3	0.3	0.3	0.3	0.4	0.5	0.4
Samples Acidified									
121	300	390	330	430	240	120	220	80	110
Bacteria Added									
142	210	240	300	260	90	13	110	62	160
175	30	55	10	320	65	1.5	2.2	2.4	3.4

TABLE 6.4 - Nickel Concentration (ppm) vs Time and Calcium Phosphate Concentration

Time (Days)	0 mg (control)	Ca(HPO ₄).H ₂ O Concentration							
		200 mg		600 mg		1000 mg		2000 mg	
		A	B	A	B	A	B	A	B
0	3	.6	.9	1.5	1.4	2.5	3	1.4	1.6
30	2.2	2.6	2	4.5	6.5	6.6	7.1	8.3	8.4
Samples Acidified									
121	680	630	520	690	740	500	530	520	640
Bacteria Added									
142	810	885	1114	1890	1285	900	1120	1050	1020
175	540	540	660	1120	860	530	690	620	920

FIGURE 6.1 - pH Data



After acidification, it was interesting to note that the control and 200 mg/L phosphate flask pH's remained stable, while the other flask pH's were much more unstable, suggesting possible dissolution and reprecipitation occurring.

DISCUSSION: The initial pH of the samples decreased with increasing

calcium phosphate concentrations. This is probably due to the formation of phosphoric acid and precipitation of CaSO_4 . The increased acid required to reach pH 3 associated with increased calcium phosphate is also probably due to a buffer effect of calcium phosphate between pH 4.6 and 3.0.

Unfortunately, despite identical procedures, ore source and time frame, the control flask did not indicate bacterial activity in this experiment as it did in Experiment #5. The reason for this is not known. The control flask bacteria may have been destroyed by pH shock, contaminated ore or unusually high and toxic copper concentrations. The interpretation of results regarding the impact of calcium phosphate on bacterial oxidation therefore, is not conclusive. However, the following trends regarding the impact on chemical oxidation were observed, and the implication of bacteria are discussed.

As expected, the acidification resulted in increased concentrations of nickel, copper and ferric iron in all flasks. The data suggested that the flasks with increasing calcium phosphate concentrations had decreasing iron and copper concentrations. This may be due to metal phosphate precipitates coating the sulphide particles, preventing dissolution. No trends were observed between nickel and calcium phosphate.

As stated earlier, the viability of the bacterial inoculation is in question. However, nickel concentration did increase significantly after inoculation relative to the control flask. There was no correlation with calcium phosphate concentration. On the other hand, ferric iron and copper concentration decreased after inoculation, with lower iron and copper concentrations associated with high calcium phosphate concentrations.

It is therefore difficult to make clear statements regarding the impact of calcium phosphate on bacterial and chemical oxidation of nickel, copper and iron sulphides. Sulphate-phosphate chemistry is complex. The presence of viable or nonviable cells and their impact is unclear. It appears that phosphate precipitates do reduce iron and copper dissolution. These precipitates may also be resistant to both low pH and the type of impact found in reciprocating shakers. Further study is warranted in column leach experiments.

EXPERIMENT #7 - Evaluation of Biological and Chemical Inhibitors on the Oxidation of Nickel-Copper-Iron Sulphide in Leaching Columns.

.....

PURPOSE:

To evaluate and compare the effects of lime, calcium phosphate monobasic, apatite, sodium lauryl sulphate, and Promac (slow release bactericide) on the oxidation of nickel, copper and iron sulphides.

METHOD:

Columns prepared from 6" PVC pipe were constructed at Falconbridge Limited. The columns were approximately 32" in length and were fitted with a cap and nipple with a clamp for drainage. A stainless steel screen was fitted over the outlet to retain the fines. An initial titration of crushed ore samples was carried out to determine the acid demand to achieve a pH of approximately 2.5, deemed ideal for bacterial leaching. Fifty grams of ore were placed into two 250 ml Erlenmeyer flasks and 100 ml distilled water was added. The samples were titrated over a three day period with 1.94 N standardized H_2SO_4 solution until apparent pH stability had been achieved. The results suggested that an acid demand of 0.15 ml of 1.94 N H_2SO_4 per gram of ore was required (0.0003 equivalents per gram of ore).

The 16 columns were loaded with 15 kg of ore on November 20. On November 25, the columns were acidified according to the amount of acid indicated by the titration results (i.e. 0.0003 equivalents/g ore or 4.5 equivalents/column). This involved the addition of 125 ml of concentrated H_2SO_4 to approximately 3 L of tap water and slowly adding this to the columns. Three liters completely immersed the ore in the columns.

The columns remained immersed for approximately two hours after which they were drained. Some H_2S was released when they were drained. After draining, the columns were

loaded with 2.5 L of pH 1.8 tap water. Acid was added daily, as necessary, in order to obtain a stable pH below 3.0. By December 16, pH stability had been established and the columns were inoculated. The columns ultimately required 0.00046 equivalents/g ore or 0.23 ml of 2N H_2SO_4 /g ore. This was 33% higher than the titration experiment had indicated, probably due to insufficient reaction time for the initial acid addition.

One hundred liters of Thiobacillus ferrooxidans culture (9k medium) were prepared between November 25 and December 9. Using centrifugal harvesting procedures, the cells from the 100 L batch of 9k were collected on December 9. The stock suspension that resulted had an optical density of 14.76. The columns were drained on December 16 and inoculated on December 17. Each column received 12 ml of the stock culture which was added to 3.0 liters of pH 2.5 tap water. A most probable number (MPN) calibration curve developed for a previous study indicated that with a harvest Fe^{+++} concentration of 7700 ppm, the corresponding MPN/ml would be 1.0×10^8 cells/ml, or 8.3×10^{12} cells. As the stock suspension had a volume of 219 ml with an O.D. = 14.76, therefore the stock suspension titre was $8.3 \times 10^{12} = 3.8 \times 10^{10}$ ml. Presumably each column received approximately 4.6×10^{11} cells or 3.0×10^7 cells/gram of ore.

Column #8 was not inoculated and served as an uninoculated

control. This column received 3 L of 2.5 pH tap water. Column temperatures averaged 19-20°C. The columns remained flooded for 48 hours until draining on December 19. The columns were not touched until January 3, 1986 at which time a 1" simulated rainfall was performed on each column. This involved the slow addition of 462 ml of pH 6.2 tap water to the top of each column. The column effluent was collected (no filtering or centrifugation). An Fe^{+++} analysis and pH measurement was performed immediately for each column effluent.

Samples for nickel and copper were stored at 4°C until atomic absorption analysis was performed at Falconbridge Limited.

After six rainfall events, the pH remained in the 2.9 to 3.4 range and nickel concentrations were elevated. While this suggests bacterial activity, the normal indicator of bacterial growth, Fe^{3+} , remained low. This phenomenon was also seen in the field. This is thought to be due to the alkalinity in the rock resulting in precipitation of iron within the column. It was thought that if the dormant period between rainfall events was doubled, that the increase in acid generated would decrease the pH to a low enough level to keep the iron in solution.

No increase in iron in the subsequent rainfall events was realized. It was therefore decided to evaluate total

acidity, rather than simply pH, and to discontinue iron analysis.

On March 31, after a total of 11 rainfall events (12 weeks), it was decided that stable concentrations of metal and acid were being generated in the column. Fourteen columns were selected from the 16 columns and received the following treatment.

Column	Reagent	Addition Rates Tons/Acre	Mg/Kg
1 & 2	Lime	5 Tons/Acre	5,450.
3 & 4	Calcium phosphate monobasic	15 Tons/Acre	16,350.
5 & 6	Lime and phosphate	1.25 Lime, 15 Phosphate	
7 & 8	Apatite	5 Tons/Acre	
11 & 12	S.L.S.	460 lb/Acre	250 mg/kg
13 & 10	Control - water only		
15 & 16	Promac	460 lb/Acre	250 mg/kg
9 & 14	Unused		
*Tons/Acre			

RESULTS: After 11 rainfall events, the 14 best leaching columns were generating the following average concentrations:

Nickel	608.4
Copper	26.3 mg/L
pH	3.18
Total acidity (to pH 8 endpoint)	3.25 gm/L
Iron	105

With the exception of iron, all analyses indicated the occurrence of bacterial growth with associated metal and

acid release. Iron analysis was expected to be at least an order of magnitude greater, but it appears that the iron is precipitating in the columns. Iron analysis was therefore discontinued.

Unfortunately, the control column was contaminated, with elevated metal and acid generation due to an active bacterial colony. Background data for evaluation of chemical oxidation rates were obtained from similar columns in Experiment #9. After a one week dormant period, four sterile columns were flushed with water, as in the column leach experiment. The average nickel concentration in the sterile controls was 130 mg/L vs 608 in the inoculated columns. Iron concentration averaged 37 mg/L vs 150 mg/L in the inoculated columns. The pH of the sterile columns averaged 0.65 pH units above the inoculated columns.

To date, there is insufficient data to evaluate the effectiveness of the treatments on the columns. Weekly rainfall events and sampling for nickel, copper, pH and total acidity will continue.

TABLE 7.1 - Column Leach Study pH Summary

.....																
COLUMN NUMBER																
Date	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Nov. 26	2.01	2.64	2.73	2.15	2.74	2.15	2.03	2.49	3.29	4.03	3.02	3.13	2.36	2.12	2.40	2.50
27	3.73	3.42	3.80	3.47	3.76	3.62	3.52	3.79	3.77	3.04	2.44	2.50	4.60	2.76	2.34	2.89
28	3.79	2.89	3.81	3.56	3.89	3.70	3.02	3.84	2.91	2.73	3.05	4.68	2.91	4.32	2.79	4.56
Dec. 2	3.96	3.24	3.79	3.90	3.98	3.90	3.66	3.86	3.05	3.47	4.03	3.86	3.28	4.05	leaks	4.18
4	3.77	2.94	3.50	3.16	3.02	3.70	2.64	3.64	3.33	3.45	3.77	3.17	3.20	3.01	-	3.09
5	3.29	2.38	2.62	2.38	3.06	2.90	2.60	2.95	2.68	2.54	2.46	2.37	2.81	3.14	-	3.11
6	3.19	3.68	3.64	3.41	3.52	3.69	3.59	3.04	3.28	2.83	3.42	3.04	3.36	3.17	-	3.73
9 all	3.84	3.80	3.81	3.66	3.53	3.90	3.42	3.88	3.68	3.19	3.82	3.34	3.60	4.02	-	3.76
10	2.32	2.29	2.18	2.07	2.16	2.62	1.96	3.30	2.14	2.03	2.16	1.98	2.15	2.44	-	2.36
11	2.37	2.21	2.32	2.17	2.43	2.54	2.08	2.13	2.51	2.03	2.44	2.14	2.15	2.94	-	2.70
12 21C	2.38	2.04	2.28	2.41	2.22	1.94	1.94	2.26	2.47	2.05	3.04	2.18	2.05	2.44	-	2.10
13 19C	leaks	2.55	2.56	2.14	2.68	2.02	2.16	2.19	2.58	2.51	2.30	2.64	2.17	2.27	-	2.54
15 20C	-	2.86	2.54	2.70	2.70	2.52	2.77	3.48	2.74	3.04	3.25	2.65	2.99	3.28	-	2.73
16 20C	2.25	1.99	1.86	1.86	1.78	1.81	1.84	1.82	1.91	1.87	1.92	1.77	1.80	1.86	2.00	1.83
18	3.53	2.81	2.83	2.96	3.63	3.05	3.15	3.82	3.42	3.36	3.53	2.84	3.23	3.25	3.76	3.45
19 19C	3.06	3.13	3.27	3.18	3.44	3.46	3.43	3.38	3.39	3.25	3.22	3.08	3.23	3.24	3.70	3.48
Jan. 3 1	3.71	4.01	3.68	3.82	3.75	3.64	3.95	3.96	3.62	3.78	3.60	3.75	3.95	3.67	4.02	3.89
10 2	3.22	3.56	3.28	3.23	3.30	3.40	3.68	3.54	3.51	3.43	3.43	3.45	3.60	3.52	3.76	3.53
16 3	3.07	3.33	3.13	3.12	3.35	3.22	3.45	3.37	3.37	3.31	3.24	3.21	3.37	3.36	3.37	3.40
23 4	3.28	3.25	3.12	3.15	3.22	3.30	3.45	3.26	3.36	3.28	3.28	3.27	3.35	3.31	3.07	3.39
30 5	3.22	3.17	3.01	2.89	3.16	3.11	3.33	3.17	3.21	3.07	3.11	3.08	3.10	3.09	2.79	3.15
Feb. 6 6	3.19	3.21	3.11	3.11	3.20	3.18	3.45	3.26	3.35	3.21	3.31	3.26	3.24	3.27	2.86	3.29
13 7															2.87	
27 8	3.05	3.35	3.27	3.17	3.30	3.33	3.58	3.32	3.49	3.33	3.31	3.36	3.36	3.33	2.88	3.24
Mar. 12 9	3.14	3.42	3.35	3.25	3.42	3.43	3.64	3.53	3.51	3.40	3.41	3.49	3.33	3.48	3.03	3.39
19 10	3.21	3.51	3.33	3.16	3.45	3.36	3.67	3.45	3.45	3.31	3.48	3.48	3.26	3.42	2.97	3.41
26 11	3.32	3.24	3.18	3.11	3.27	3.15	3.42	3.18	3.32	3.14	3.26	3.27	2.99	3.24	2.88	3.22
	Line		CaPO ₄		CaPO ₃ +Line		Apatite		Nothing C		S.L.S.		C Nothing		Promac	
Apr. 8	3.60	4.04	2.79	2.87	2.88	3.13	3.48	3.35	3.31	3.15	3.70	3.70	3.12	3.31	3.23	3.48

TABLE 7.2 - Column Leach Study Iron Summary

.....																
C O L U M N N U M B E R																
Date	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Dec. 14	Composite Fe+++ = 93 ppm															
Jan. 3	84	68	155	145	107	115	64	82	69	42	154	92	54	62	86	63
10	120	86	128	122	103	107	50	73	122	50	73	63	42	62	69	56
16	141	122	124	148	94	88	48	133	85	109	114	127	102	98	183	85
23	126	83	139	157	88	93	58	137	73	83	93	79	80	89	201	62
Total	912	78	151	442	95	128	67	121	121	67	316	621	80	190	355	197
30	134	128	149	137	101	109	76	120	53	73	76	68	54	66	221	56
Feb. 6	153	133	145	149	113	113	107	130	68	96	83	102	96	80	350	79
13	Extended Dormant Period															322
27	150	118	110	122	132	111	85	126	72	97	102	86	123	76	220	86
Mar. 12	158	76	104	178	101	77	67	114	36	68	73	74	100	85	222	63
19	DISCONTINUED															

TABLE 7.3 - Column Leach Study Cu Summary (ppm)

.....																
C O L U M N N U M B E R																
Date	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Jan. 3	95	60	138	104	90	87	54	128	51	39	182	91	46	54	75	46
10	78	62	145	112	87	76	36	99	86	36	44	37	36	50	104	30
16	113	112	160	135	100	95	57	167	46	46	101	75	42	44	210	37
23	84	89	138	100	145	104	68	165	27	42	59	48	32	36	140	29
30	64	48	100	100	57	49	42	77	23	35	44	38	32	31	157	25
Feb. 6	74	54	119	59	48	39	50	69	18	30	33	28	25	21	230	32
7	Extended Dormant Period															220
27	91	65	111	126	65	55	39	260	17	38	54	31	40	26	176	37
Mar. 12	Total Acid Only (2 Week Dormant Period)															
19	42.6	47.3	55.8	52.2	38.7	24.8	17.7	35.7	6.4	15.9	19	12.2	24.7	8.7	72	9.4
26	30	59	47	35	37	19	17	29	5	13	14	9	29	7	40	6

TABLE 7.4 - Column Leach Study N1 Summary (ppm)

.....																
C O L U M N N U M B E R																
Date	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Jan. 3	570	227	282	290	328	268	221	330	296	155	500	255	220	398	480	372
10	650	445	430	430	360	325	221	364	322	258	370	147	262	423	645	405
16	572	465	482	465	350	420	292	514	389	250	340	320	300	376	1040	423
23	595	407	450	515	525	515	430	544	353	240	370	385	348	360	360	432
30	522	525	529	459	322	506	500	606	318	222	230	260	330	360	695	424
Feb. 6	570	670	615	600	385	620	690	710	430	315	400	340	452	469	805	563
13	Extended Dormant Period															805
27	600	680	540	633	500	660	650	680	340	364	380	367	530	465	640	462
Mar. 12	Total Acidity Only															
19	700	770	680	615	655	720	600	745	350	415	500	360	465	415	480	530
26	870	800	620	620	600	780	600	760	350	455	510	360	420	380	645	470

TABLE 7.5 - Column Leach Study Total Acidity

.....																
C O L U M N N U M B E R																
Date	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Mar. 13	3.45	3.78	4.19	3.51	4.96	3.83	3.40	5.14	1.53	2.21	3.02	2.45	5.28	1.49	4.60	2.44
20	3.40	3.44	3.30	2.85	5.15	3.10	2.81	3.71	1.13	1.73	2.93	2.67	2.56	2.97	2.53	2.61
28	4.43	3.61	3.04	3.01	5.02	3.28	2.70	3.53	1.21	2.27	2.56	2.30	3.46	1.21	3.99	1.95

EXPERIMENT #8 - Buffering Capacity of Strathcona Mill Ore.

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INTRODUCTION: From Experiment #5, it became obvious that a considerable alkalinity was present in the ore, despite the high sulphide content. It was apparent that ore kept under water had a relatively stable pH, probably due to the oxygen barrier created by the water cover, preventing oxidation of the sulphides. The following experiment was completed to evaluate ore alkalinities to various pH endpoints.

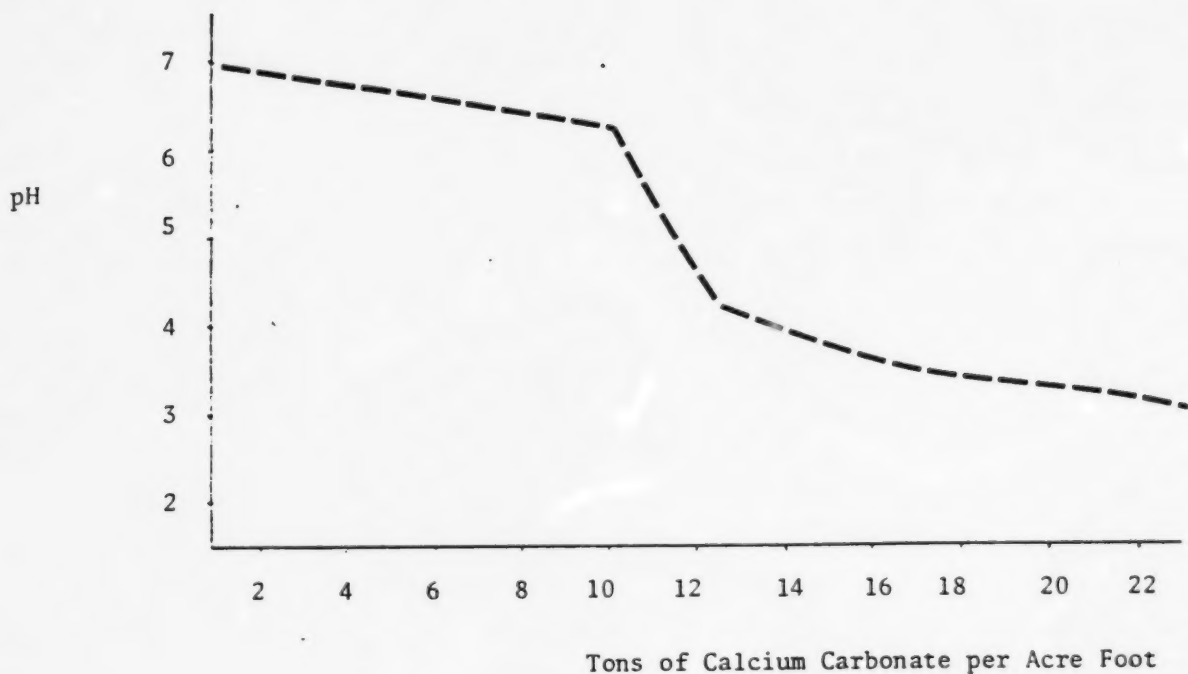
METHOD: Twenty-six 25 gr samples of screened (-20 +28 mesh) Strathcona Mill feed were slurried with 50 mls of distilled water. Increasing amounts of sulphuric acid were added to the beakers and left to stabilize for several days.

RESULTS: Results of this research are summarized in Figure 8.1. From this graph, it can be seen that to a pH 3 endpoint, the ore alkalinity is approximately 23 tons CaCO_3 /acre foot when the ore is kept under water. This is due to the lack of oxidation of the sulphide due to the oxygen barrier created by the water. Therefore, shake flask studies are of limited value in evaluating ore oxidation. Column leach experiments are of greater value as they more closely model the field situation, permitting oxygen penetration into the column.

FIGURE 8.1 - Alkalinity of Strathcona Mill ore
.....

Figure 8.1

ALKALINITY OF STRATHCONA MILL ORE



EXPERIMENT #9 - Improvement of Sodium Lauryl Sulphate
Persistence

.....

INTRODUCTION: The most serious drawback to the use of sodium lauryl sulphate (S.L.S.) for the control of acid mine drainage and metals release is its relatively short persistence. The best results with S.L.S. occurred in coals spoils where it was theorized that S.L.S. was adsorbed by carbon, improving its persistence. Since there was little carbon available in Falconbridge waste, attempts were made to improve S.L.S. persistence utilizing other sources of waste carbon. Attempts were also made to bind S.L.S. to the sulphide particles with concentrator flotation reagents.

PURPOSE: To evaluate the ability of waste sawdust, coke fines, mulch and flotation reagents to improve the persistence of sodium lauryl sulphate for the control of acid mine drainage and metals release from waste rock.

METHOD: An S.L.S. ore absorption test was completed (Appendix A) from a 1.06 gr/L stock solution of S.L.S.

Based on the absorption test, leach column tests were set up. Approximately 0.3 gr of S.L.S. (20 mg/kg) in various formulations were added to 32" PVC columns filled with 15 kg of Strathcona Mill ore (as in Experiment #7). The following formulations were evaluated, based on the

maximum capacity of the hydroseeder:

<u>Column</u>	<u>Treatment</u>
1	0. grams SLS (approximately 20 mg SLS/kg)
2	0.3 grams SLS + 10 grams coke fines
3	0.3 grams SLS + 10 grams sawdust
4	0.3 grams SLS + 10 grams commercial mulch

The stability and potential bond formation of a S.L.S. - Xanthate flotation reagent mix was also researched.

Following the reagent addition, twenty, one inch rainfall events were simulated by slow additions of 400 mls of distilled water to the columns over a two week period. The volume and concentration of S.L.S. in the effluent was analyzed after each event.

RESULTS: The S.L.S. adsorption capacity of the ore utilized in the column leach experiment was 15.6 mg/kg/ore. The endpoint of this test occurred at 10.6 mg/kg S.L.S., or 2-1/2 times the bactericidal concentration of S.L.S. observed in Experiment #2. Based on this result, it was decided to evaluate 0.3 gr. of S.L.S./column, or 20 mg S.L.S./kg ore.

The results of the rainfall events are summarized in Figure 9.1 and 9.2. The control columns released toxic concentrations (.4 mg/L) of S.L.S. for 14 rainfall events, while the treated columns released toxic

concentrations for 13 events. After subtracting the ore adsorption of S.L.S. of 15.6 mg/kg, the percentage of S.L.S. released after twenty rainfall events ranged from 45% to 81%. As expected, coke fines adsorbed the greatest amount of S.L.S., followed by the mulch, with the sawdust and control columns relatively equal. (Table 9.1)

Despite the adsorption of S.L.S. by the coke, the bond between the S.L.S. and coke was too strong such that concentrations of S.L.S. released from the coke were too low to be toxic after 13 rainfall events. Therefore, it is not recommended that S.L.S. be mixed with any of the wastes tested.

Research into utilization of S.L.S. and Xanthate to bind S.L.S. to the active sulphide sites at first appeared promising. The potential for a weak S.L.S.-Xanthate bond existed. However, the Xanthates themselves were not stable. Under acidic conditions, Xanthate ($C_5H_{11}'O'CS'SNa$) rapidly decomposed. Even under alkaline conditions it decomposes at a rate of approximately 10%/week. Therefore, attempts to bind S.L.S. to the sulphides with Xanthate would be impractical.

TABLE 9.1 - S.L.S. Released in Leaching Columns

.....

		S.L.S. & <u>Coke</u>	S.L.S. & <u>Sawdust</u>	S.L.S. & <u>Mulch</u>
S.L.S. released after 20 rainfall events	17.4%	10.2%	19.4%	16.2%
S.L.S. released at bactericidal concentrations (4 µg/L)	16.3%	9.3%	18.3%	14.9%
S.L.S. released after subtraction of ore adsorption (15.6 µg/kg)	79.8%	45.4%	85.7%	73.6%
S.L.S. released at bactericidal concentrations after subtraction of ore adsorption (15.6 µg/kg)	74.9%	41.2%	80.7%	67.6%

FIGURE 9.1 - Concentration of S.L.S. Released in Rock Columns

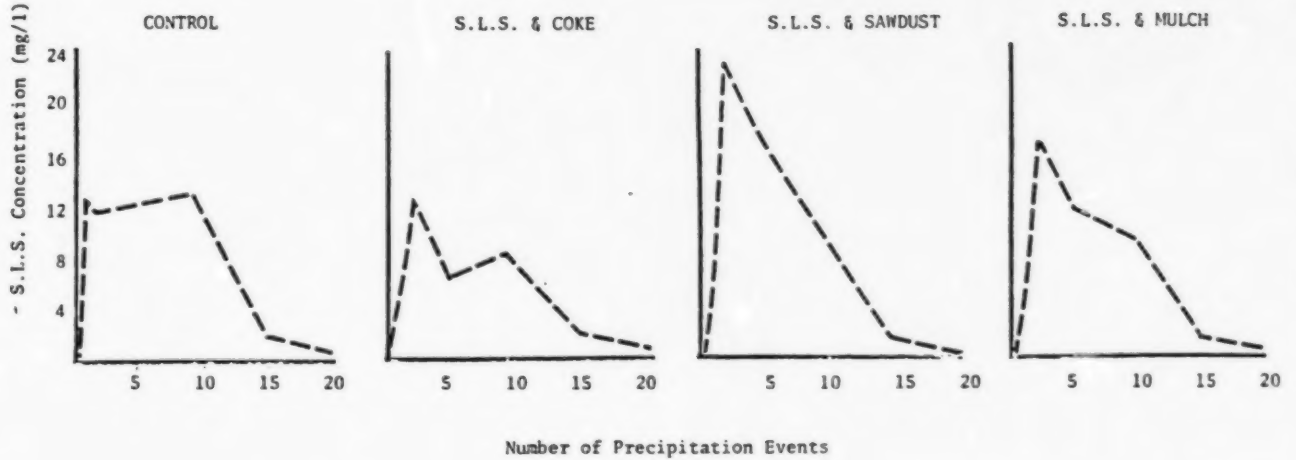
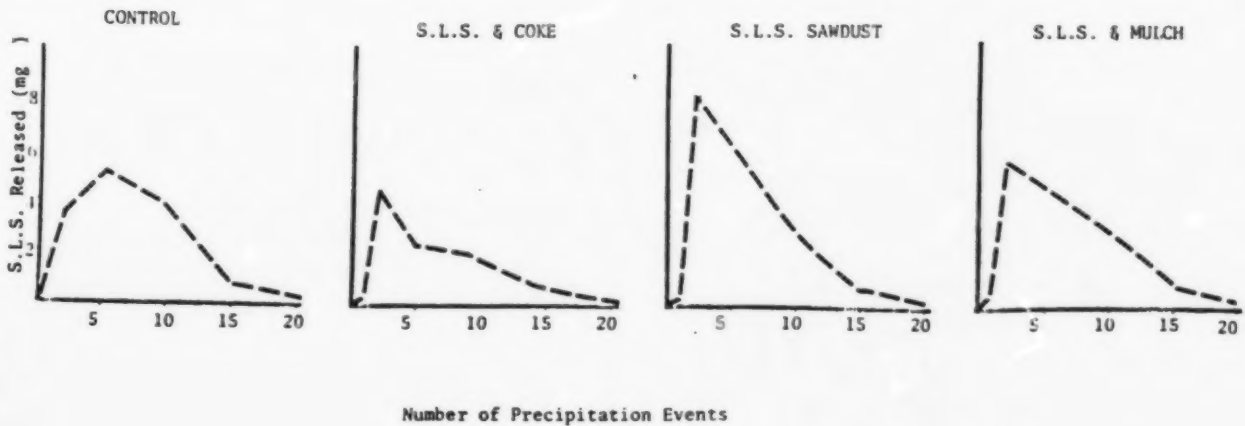


FIGURE 9.2 - S.L.S. Released in Rock Columns

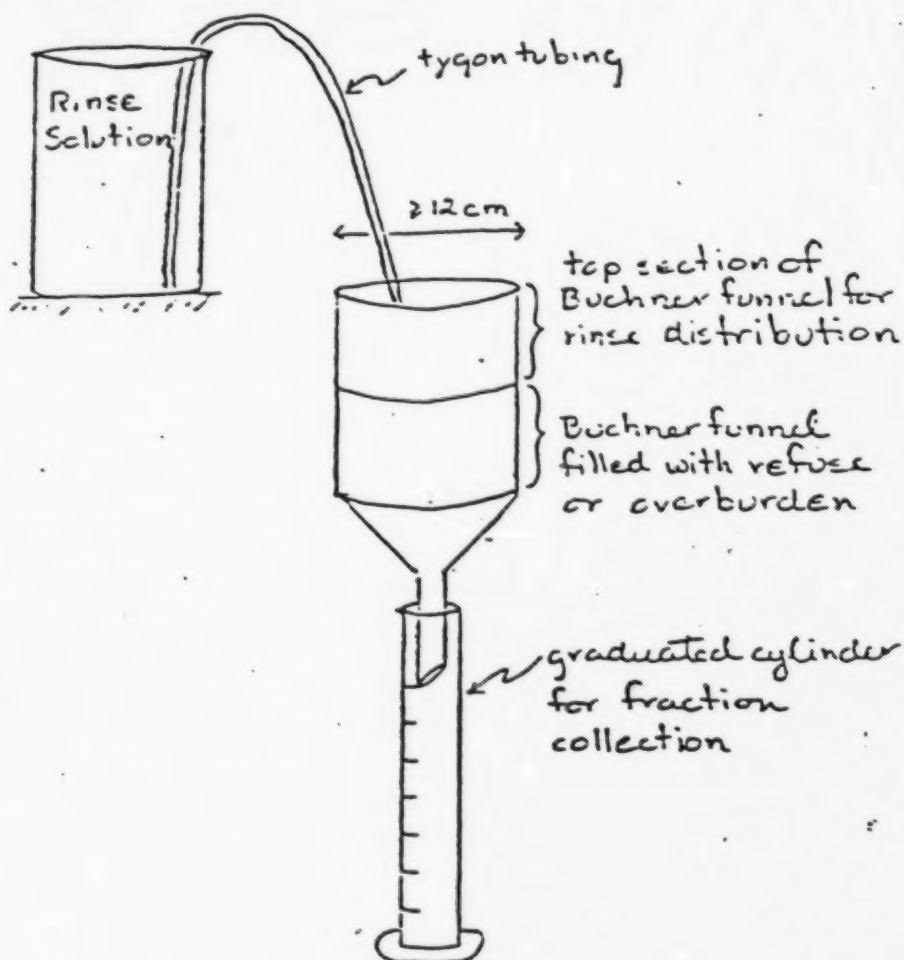


APPENDIX A

SLS ADSORPTION TEST

The test described below is a method for estimating detergent adsorption on coal, refuse and overburden. We are currently using this procedure for determining SLS application rates for field tests.

Test Set-Up:



Procedure:

1. Place filter paper in Buchner funnel top and weigh. Pack with material to be tested to 1 inch or 2 inches in depth and obtain weight of material by difference.
2. Calculate the amount of SLS needed for a treatment level of 25 mg SLS/kg sample for refuse; 40 mg SLS/kg sample for coal or spoil. Prepare a stock solution of SLS, in the concentration range 300 to 3,000 mg/l.

Distribute the appropriate volume of solution evenly over the sample surface.

APPENDIX A - Cont'd.

3. Fill the rinse reservoir with distilled water and place as shown above. Start rinse by applying suction to tubing with a pipet bulb. Flow rate through the material is usually determined by grain size; use a screw clamp on tubing to regulate rinse flow to minimize ponding on material surface. Collect rinses in 100 ml fractions in a graduated cylinder, until effluent does not form suds on shaking.
4. Analyze stock solution and collected fractions for surfactants by the methylene blue method (APHA Standard Methods) or equivalent procedure.
5. Calculate the weight of SLS adsorbed as the difference between maximum weight recoverable and actual weight recovered:

$$\text{SLS adsorption capacity} = \frac{C_s V_s - \sum_{i=1}^n C_f V_f}{\text{sample weight}}$$

Where: C_s = mg SLS/l in stock solution

C_f = mg SLS/l in collection fraction

n = Number of fractions collected

V_s = Volume of stock solution applied, in liters (step 2)

V_f = Fraction volume

Express adsorption as mg SLS/kg material or lb SLS/lb material.

6. Calculate the amount of SLS needed to treat the upper 1 foot of material at the site:

SLS adsorption capacity x density of material x site area

(lb SLS/lb sample) (lb/acre-foot) (acres)

Notes on Procedure:

- A. Some materials are relatively impermeable and ponding will not be avoidable. Long flow through times are typical in these materials.
- B. If the application rate results in nearly complete adsorption or minimal adsorption, repeat the test with a more appropriate SLS solution.

GARBAGE AND WASTE ROCK TEST PLOTS AND
WASTE ROCK RECLAMATION EXPERIMENTS

The Fecunis tailings pond test plots are approximately 90% complete, despite the late start, limited waste availability, and early 1985 snowfall. The plots are 8000 sq. ft. by four feet deep (Figure 1) and run parallel to the surveyed surface runoff. One plot is lined with a tailings fill and plastic liner. The other two will be completed shortly to eliminate cross contamination. Surface drains are installed.

Rather than install sand points for collection of ground water, it was decided to install vacuum tensiometers. Two will be installed in each plot, one in the hardpan, one below it (approximately 1 ft. and 3 ft. deep respectively).

As soon as the tensiometers can be installed, the waste rock and garbage will be placed in the plots. Waste rock, garbage, and plot liners will be completed with Falconbridge funding. Sampling should begin in July, 1986.

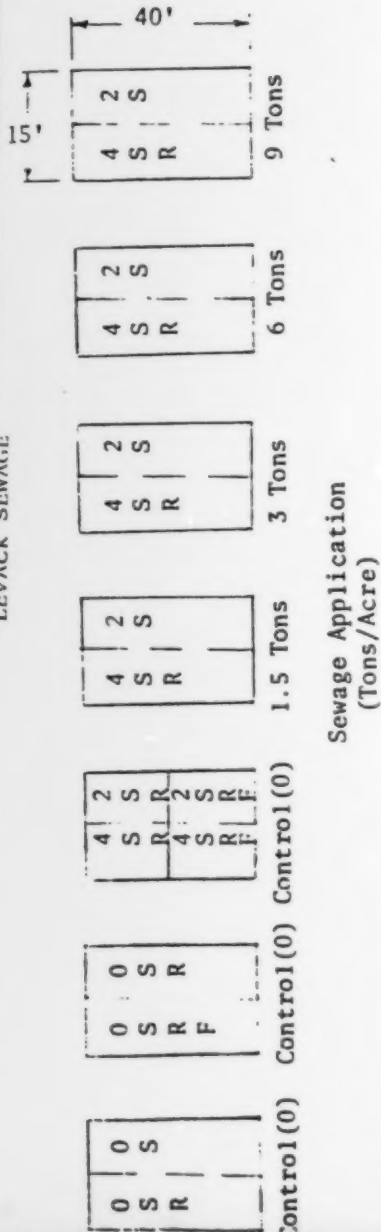
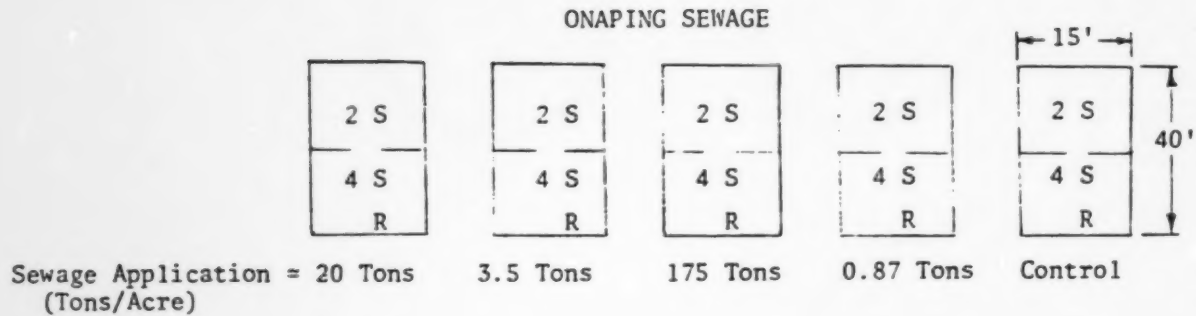
The following is submitted for your information, but was not part of the 1985 research proposal. However, this research does have a direct bearing on the 1986 work and is therefore included.

During parts of 1984, 1985 and 1986, approximately 70 acres of the Fecunis tailings pond has been covered with 4-6 ft. of waste rock (850,000 tons). Of this 70 acres, roughly 35 acres have received approximately one ton/acre digested sewage sludge.

The waste rock contains an average 0.13% nickel, 1.7% iron, 0.008% copper, 1.2% sulphur. The total sulphide concentration is approximately 3%. The sewage was added as a 1.5-2.5% solids slurry, containing 0.52 mg/L phosphorous 1.4 mg/L Kjeldahl nitrogen, 0.2% ammonia (as N) and a five day B.O.D. of 3.4 mg/L (as O).

Twenty-four test plots were established in 1985 in order to evaluate the optimum sewage addition rate and required lime or fertilizer applications (Figure 10.1). Growth on the plots will be evaluated in late May - early June, 1986.

FIGURE 10.1 - Waste Rock Reclamation Test Plots



FECUNIS TAILINGS POND

KEY:

- R - Rye seed at 60 lb/acre
- S - Grass seed at 80 lb/acre, containing
 - 20% Creeping Red Fescue
 - 20% Red Top
 - 15% Tall Fescue
 - 15% Timothy
 - 15% Canada Blue Grass
 - 10% Alsike Clover
 - 5% Birds Foot Trefoil

F - Fertilizer at 300 lb/acre 10-10-10

0 or 2 or 4 - Limestone application in tons/acre

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DEVELOPING A TECHNIQUE FOR RECLAMATION
OF HIGH IRON SULPHUR TAILINGS BY
ESTABLISHING MARSHLAND

Work on this project was conducted under the auspices of the Canada
Centre for Mineral and Energy Technology, Energy, Mines and
Resources, Canada

DSS File Number: 23 SQ.23440-5-9163
DSS Contract Serial Number: OSQ85-00154
Canmet Project Number:
Canmet Scientific Authority: Dr. N. Dave

EXECUTIVE SUMMARY - 1985

PROJECT 1 - Developing a Technique for Reclamation of High Iron Sulphur Tailings by Establishing Marshland.

Objectives:

1. Maintain a water cover over pyrrhotite area to eliminate or reduce oxidation.

Seepage water would be monitored to determine the impact of the water cover in impeding oxidation. Due to the late start of this project the seepage sampling would not be conducted until the second season. A field lysimeter approach to monitor the seepage will be performed in order to eliminate cross contamination between the respective test cells.

2. Identification of suitable seed-root materials for transplanting.

By incorporating aquatic plants with the water cover, organic material will accumulate over the tailings with time. Eventually a thick organic layer will result such that anoxic conditions may result at the organic-tailings interface without the water cover. At this stage, the tailings area reclamation would be complete and a walk away situation would exist.

Information was therefore required in:

- a) determining which plant species grow in this environment.
- b) rate of spread and organic buildup.

c) effect of organic buildup on oxidation of tailings.

3. Methodology for transplanting or initiating plant growth.

A method that is cost-effective in producing an aquatic plant cover over a large area had to be evaluated. By constructing large test plots (1/3 acre) then a proper costing of machinery, labour, equipment and power requirements may be made. Appropriate amelioration with lime, fertilizer, sewage or combinations of the above must be evaluated. Methods of aquatic plant establishment, also have to be considered, i.e.: seeds vs root sections vs whole plants; time of planting; equipment required; seed pre-treatments required.

Test Plots:

1. Thirty holes, 3-4 feet deep and 3 feet in diameter, were dug out of the pyrrhotite, lined with 10 ml polyethelene and refilled with excavated material. These would serve as small test plots to evaluate:
 - a) suitable plant material.
 - b) time of planting.
 - c) effect of various treatments (sewage, lime, fertilizer).
 - d) method of planting (seeds, rhizomes, whole plants).

Due to the late start of this project these test plots were to be initiated in the second season. Problems arose in establishing these plots, when the holes were dug and fresh pyrrhotite (Po) was exposed to the atmosphere. The oxidation of the Po caused

spontaneous combustion in some of the plots, which burned holes in the plastic liners. A field lysimeter approach is therefore proposed as a more feasible method for establishing these small plots (refer to second submission - April, 1986).

2. Three large test cells (~1/3 acre each) were constructed by building 5' high gravel berms and sealed with fine grained material. A Gordon Rupp pump (6 cylinder, 426 cu. in. Detroit diesel) was utilized to pump ~160 gallons per minute from a small spring fed pond (1 acre) into each test cell. The test cells were ameliorated as follows:
 - a) 2 ton hydrated lime sprayed by hydroseeder into cell #3.
 - b) septic tank sewage dumped into cell #2 (still in progress).
 - c) control cell - no treatment.

As with the small test plots, useful information was gained during the short time period of operation (one month). The oxidized layer of Po had formed a fragmented hard pan which was highly permeable. Maintaining a perched water table was therefore difficult with the situation as it existed. For a large scale project a more expensive deep well would have to be considered. Sealing the Po surface with lime or sewage sludge was therefore considered as a viable alternative to relocating the water supply.

Future proposed work will downsize these test plots by half and incorporate 1 to 2 feet of organic matter to accelerate the reclamation process and prevent oxidation of the Po.

Preliminary Results:

Outlined below is a brief summary of the seedling establishment (lab and field) work conducted by Boojum Research for Falconbridge Limited. For a more detailed report refer to the appended document "Developing a Technique for Reclamation of High Iron Sulphur Tailings by Establishing a Marshland, Phase 1, Selection of Suitable Plant Material and Methods of Establishment".

1. Percolation

- a) Three holes utilizing 30" diameter ABS piping were constructed in the Po-tailings and filled with 30 gallons of water. All three were dry in 4-5 hours. This high permeability is attributed to the hard pan formation which is broken up into aggregated particles by frost action. Large air pores therefore exist in the oxidized zone (2-3 feet thick) making it highly permeable. The hard pan is also layered, similar to a shale, and water movement between these layers by capillary action would be relatively rapid (field observation only, not measured).
- b) The large test cells were flooded to varying degrees:
 - i) 100% water cover 1' deep in cell #3.
 - ii) 80% water cover 8" deep in cell #2.
 - iii) 50% water cover 4" deep in cell #1.

Insufficient water in the spring fed pond resulted in the pump

repeatedly losing its prime. After 3 to 4 days, with no pumping, all ponds would be dry.

2. Vegetation

- a) 10 cattails were transplanted in the tailings section (west of the test cells, adjacent to the dam). Two appeared alive and only one had a new shoot after one month. All plants appeared dead after two months - but two had new shoots.
- b) Cattail seeds were spread throughout this area but failed to germinate.
- c) A moss spread around the tailings pond perimeter was green after one month but not spreading. After two months this moss appeared to be spreading at the south side of the pond.
- d) A green algae from the Conservation area placed in the pond appeared dead.

Initial pH of the tailings pond water was 7.1 to 7.6 (July) but had dropped to 4.4 (August).

3. Chemistry (Lab)

Water was placed over Po and over tailings for:

- a) 38 days continuously. (Long term)
- b) 38 days with 3 replacements of fresh water. (Sequential)

The results are summarized in Table 1.

TABLE 1

Metal content in water covering pyrrhotite and mine slimes (ppm)
in sequential and long term leaching experiments

Metal	08 July*		23 July		09 August		Total	
	Filt. ppm	Unfilt. ppm	Filt. ppm	Unfilt. ppm	Filt. ppm	Unfilt. ppm	Metal conc. ppm	
<u>Pyrrhotite</u>								
Fe sequential	42.2	42.0	115	115	85	87.5	242	244.5
long term					344	375	344	375
Ni sequential	12.1	12.1	2.9	2.93	1.49	1.49	16.5	16.5
long term							12.0	12.0
Cu sequential	1.62	1.55	0.29	0.29	0.08	0.08	2.0	1.9
long term							0.93	0.84
<u>Mine Slimes</u>								
Fe sequential	0.06	318	0.93	1.92	0.20	23.1	1.2	343
long term							0.31	0.42
Ni sequential	2.11	12.5	0.29	0.33	0.18	2.05	2.0	14.9
long term							0.37	0.42
Cu sequential	0.03	1.92	0.29	0.03	0.02	0.31	0.34	2.26
long term							0.02	0.03

*Experiment began on 3 July 1985.

4. Seed Germination

- a) Various seed species (cattails, reed grass, rushes) were subjected to different treatments to determine how germination is affected. The results are summarized in Table 2.
- b) Bromus sp. and Scirpus sp. were successfully germinated directly on pH 7.0 and 10.0 amended Po. Cattail seeds germinated on amended Po but to a lesser extent (10% vs 100% in control). Also the cattails failed to grow beyond the seedling stage in pH 7.0 Po. Hand planting of whole plants therefore appears as the only alternative to slightly acidic or neutral Po.
- c) Hand planted cattails in acidic areas resulted in new shoots from the rhizomes after the initial plant had died. No seeds germinated on unamended Po (pH 2.3-2.5) or on Po amended with sewage sludge.
- d) Cattail seeds collected from an acidic environment resulted in better germination rates (38%) than cattail seeds collected from control areas (12.5%) or alkaline areas (4.7%).
- e) Scarified rush and sedge seeds did not improve germination. Freezing of seeds, also did not improve germination. Best results were obtained by air drying at room temperature.

TABLE 2

Effect of Pre-germination Protocols on Seed Germination

	Room Temp		Room Temp In Dessicator		Frozen	
	2 Wks	3 Wks	2 Wks	3 Wks	2 Wks	3 Wks
<u>SEED TYPE</u>						
Sedge Species #1	1/104 (0.9%)	1/104 (0.9%)	2/78 (2.6%)	2/78 (2.6%)	1/27 (3.7%)	1/27 (3.7%)
Sedge Species #2	0/15	0/15	0/23	5/23 (21.7%)	0/17	0/17
Sedge Species #3	0/12	1/12 (0.8%)	0/12	0/12	0/10	0/10
Rush Species #1	0/16	0/16	0/14	0/14	0/12	0/12
Grass #1	6/24 (25%)	6/24 (25%)	3/36 (8%)	3/26 (8%)	0/24	0/24
<u>CATTAILS</u>						
Acid	11/29 (38%)	-	-	-	-	-
Alkaline	4/86 (4.7%)	-	-	-	-	-
Control	9/72 (12.5%)	-	-	-	-	-